

All Redactions In This
Document Are Ex. 4 - CBI

Inhance EPA Technical Discussion

May 26, 2022 Presentation to EPA

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Introduction

- Timeline for responses to EPA questions
- Scientific validation for the process modifications implemented
- Test methods discussion

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Response to EPA questions

- Working on the written response to specifically answer questions, will submit through CDX by June 6, 2022
- Slides from today will be submitted through CDX by June 6, 2022

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Scientific Validation for the Process Modifications – Approach

- Study scope:
 - 12+ months of research
 - Hundreds of experiments
 - Methods discussion

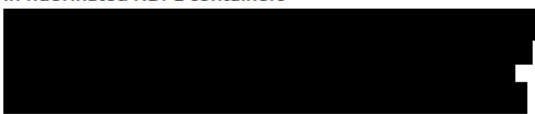
• [REDACTED]
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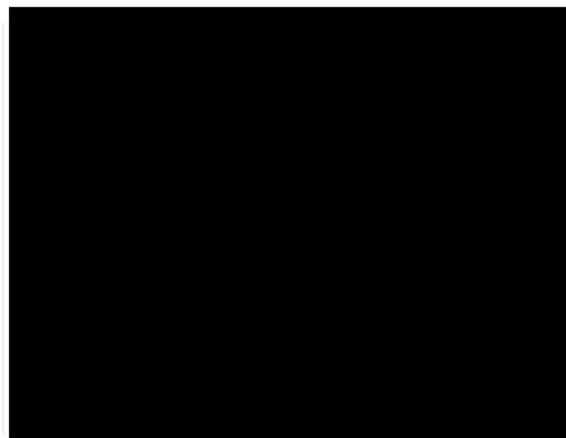
- Scientific validation of process modifications to suppress LCPFAC formation

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Influence of Oxygen on Potential LCPFAC Formation

- Eliminating oxygen from the process would be readily feasible, IF it was the root cause for LCPFAC formation. Inhance could add additional vacuum cycles to further reduce residual oxygen in the chambers.
- Multiple research papers* indicated that fluorination in the presence of oxygen could be the source of PFAS formation in fluorinated HDPE containers
- 
- Previous researchers hypothesized formation of PFAS in fluorinated HDPE without studying input variables (only outputs were measured)
- None of these research papers systematically investigated the influence of oxygen/air on the presence or formation of PFAS



*Kharitonov et al. "The kinetics and mechanism of the direct fluorination of polyethylenes" Surface Coatings International Part B: Coatings Transactions, 201, Vol. 8, pp. 137-230, September 2002; Kharitonov et al. "Direct fluorination of polymer vessels and membranes" Facta et chimique - octobre-novembre 2006 - n° 301-302; Rand et al. "Perfluorinated Carboxylic Acids in Directly Fluorinated High-Density Polyethylene Material" Environ. Sci. Technol. 2011, 45, 8033-8039

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HCA in HDPE Largely Result in LCPFAC Formation During Fluorination

- Hydrocarbon carboxylic acids (HCAs) are present in the HDPE
- These HCAs are unintentionally formed due to degradation of HDPE during extrusion*

The long-chain HCAs present in HDPE are fluorinated during the fluorination process, resulting in LCPFAC formation

- Next slide will discuss results that support this conclusion

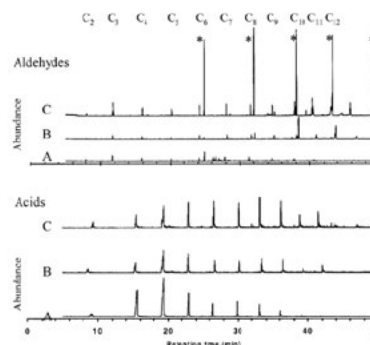


Figure 10 GC-MS chromatograms obtained from (A) LDPE, (B) LLDPE, and (C) HDPE films processed at extrusion process temperatures of 315°C and 40 mm air gap. Oligomer esters identified as decane, dodecane, tetradecane, hexadecane, and octadecane, respectively, are marked with an asterisk. Chromatogram marked as TIC=Total Ion Count, as Aldehydes=ion count $m/z=44$, and as Acids=ion count $m/z=60$.

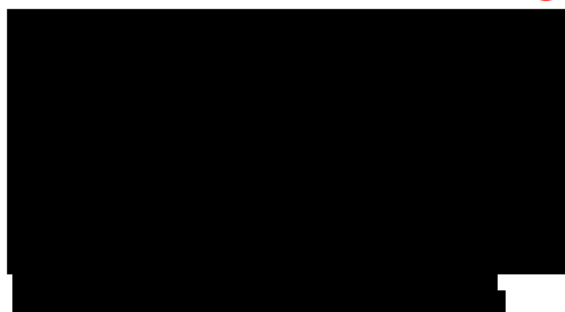
*Andersson et al. "Degradation of Polyethylene During Extrusion. II. Degradation of Low-Density Polyethylene, Linear Low-Density Polyethylene, and High-Density Polyethylene in Film Extrusion" Journal of Applied Polymer Science, Vol. 91, 1525-1537 (2004)

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Removal of Long-Chain HCAs Results in LCPFAC Reduction in Fluorinated HDPE

- No solvents known to solubilize HDPE to facilitate thorough extraction
- A reference HDPE sample was soaked in a solvent mixture of toluene and methanol (50/50 mixture) for 2 days at 50°C. Toluene was used to swell the polyethylene, while the methanol was used to extract the HCAs.
- The solvent treated HDPE was dried and fluorinated
- [REDACTED]
- [REDACTED]
- Incomplete extraction of HCAs from HDPE is the likely reason for partial rather than full elimination of PFOA



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Process Modifications Resulted in Non-Detect LCPFACs in Fluorinated 2.5 Gallon HDPE Container Cutouts

- [REDACTED]
- [REDACTED]
- [REDACTED]
- [REDACTED]
- [REDACTED]
- [REDACTED]
- Successful trial demonstrated non-detection for LCPFACs using the modified process
- CEN method and EPA method analysis demonstrated non-detect LCPFACs with the modified process

Most research and developmental work was performed on reference HDPE samples, as well as 2.5 gallon natural HDPE containers



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HDPE Composition Variability May Require Further Process Modifications

- HDPE has several additive packages that vary by HDPE manufacturer and the molder
- HDPE blow-molding processing conditions vary widely from molder to molder
- Amount of long-chain HCAs in HDPE depends on resin, additive package and processing conditions
- As we continue to analyze for LCPFACs in fluorinated HDPE, it is possible that further process modifications may be required to address specific HDPE compositions

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Accelerated Degradation of Fluorinated HDPE Shows Non-Detection of LCPFACs

- Accelerated weathering tests are often used to simulate degradation of plastics. Testing subjects plastics to UV light source which promotes breakdown of the plastic. HDPE degrades through a radical chain scission mechanism under UV, resulting in reduction of mechanical properties*
- [REDACTED]
- Results demonstrated non-detection for LCPFACs at 0 hours UV exposure, and 1500 hours UV exposure (>>> 1 year shelf-life equivalence**)
- Even with significant degradation of fluorinated HDPE in the weathering tests (complete loss of mechanical properties) LCPFACs were not detected, suggesting that degradation or peroxy radicals are not sources of LCPFACs

Species	Level 5 Sample ppb (ID 436)		Level 9 Sample ppb (ID 433)			
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]

*Graessle et al. "Changes during the weathering of polyolefins." Polymer Degradation and Stability 181 (2020) 109364

**Hill et al. "Correlation of a Temperature UV-Weathering Cycle to Outdoor Exposure for the Determination of the Environmental Instability of Polyethylene Films Using HT-GPC Analysis." Polymers (Basel). 2021 Feb; 13(4): 391.

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Guidelines for Suppression of LCPFAC Formation in HDPE

- Each of these inputs contribute towards the formation of LCPFAC impurities
- [REDACTED]
- Combination of variables can influence LCPFAC formation

[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]

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Test Methods Discussion

- EPA test method for rinsate from HDPE containers
 - Difficult to replicate across all container sizes and shapes. Volume of methanol and container size ratio cannot be established easily
 - User variability in the sloshing of the containers
 - Typically capable of lower detection limits
 - Good surrogate recoveries have been a challenge when using isotope dilution method
 - Results reported as ng/Liter (ppt)
 - Data shared with EPA for 2.5 gallon HDPE containers, demonstrating non-detection of LCPFACs using modified process
- CEN 15968:2010
 - Often used method for evaluating PFAS in plastics
 - Excellent surrogate recoveries when using isotope dilution method
 - More consistent sampling and testing regardless of container shape and size
 - More aggressive extraction protocol (2 hours, 60°C, ultrasonic water bath)
 - Results reported as µg/kg (ppb)
 - Data shared with EPA for 2.5 gallon HDPE cutouts, demonstrating non-detection of LCPFACs using modified process

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